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The Effect of Calcination Temperature on Crystal Structure of $(Al,Cr)_2O_3$ Solid Solution

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Abstract

$(Al,Cr)_2O_3$ solid solutions were synthesized using aluminium oxide (Al_2O_3) and chromium oxide (Cr_2O_3) as the starting materials. These two oxide powders were mixed at 1:1 mole ratio using ball mill. Then the mixture was dried and calcined for 2 hours at various temperatures of 1,500 °C, 1,600 °C and 1,700 °C. The structures of the prepared powders were investigated by X-ray diffraction (XRD) technique. The XRD result showed that $(Cr_{1-x}, Al_x)_2O_3$ presented as a new phase in the calcined powders for all temperatures. The XRD pattern illustrated intensity of $(Cr_{1-x}Al_x)_2O_3$ peaks slightly increased and shifted to Al_2O_3 peaks when the calcination temperature increase, while intensity of Al_2O_3 peaks gradually decreased. From this result, it can be implied that Cr^{3+} in Cr_2O_3 rhombohedral structure was gradually substituted by Al^{3+} at high temperature. However, the completed reaction was clearly shown at 1,700°C. In addition lattice parameters (*d*-spacing 311 plane. *a* and *c*) were inversing with the calcined temperature.

Keywords: XRD pattern, Calcination, Solid solution, Alumimium oxide, Chromium oxide.

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1. Introduction

Aluminium oxide (Al_2O_3) and chromium oxide (Cr_2O_3) are promising ceramic materials. Because chromium oxide is very high hardness and has a wide range of applications such as wear resistance, corrosion resistance, electronics and optics [1-5]. In addition, aluminium oxide has also many applications in mechanical strengths, and thermal and chemical stabilities. It is well known that α-Al_2O_3 is thermodynamically stable at room temperature [6, 7]. Therefore the solid solutions of aluminium and chromium oxide are interested to improve its thermal and chemical stability to use in several applications.

In the other hand, Al_2O_3 and Cr_2O_3 are having the same crystal structure with Al^{3+} and Cr^{3+}ions occupying two thirds of available octahedral interstitial sites to form Al_2O_3/ Cr_2O_3 solid solution by reaction at high temperature. The substitutions of Al^{3+} and Cr^{3+} were effected to lattice parameter of solid solution [8]. Therefore the aim of this experiment is to study the structural characteristics of substitutional solid solution of Al_2O_3/ Cr_2O_3 by solid state reaction at temperatures between 1,500°C to 1,700°C in the air.

2. Materials and experiment

The $(Al,Cr)_2O_3$ solid solution was prepared by solid state reaction. The powders of α-Al_2O_3 (0.5μm) and α-Cr_2O_3 (0.5μm) as 1:1 mole ratio were used for starting materials by mixing in distilled water by ball mill for 6 h. Then homogeneous mixture was dried in an air oven at a temperature of 105°C for 24 h. These dried powders were calcined at various temperatures of 1,500°C, 1,600°C and 1,700°C under air atmosphere with each temperature for 2 h. The calcined powders were ground by ring milling to reduce particle size. Phase identification was performed by X-ray diffractometer (Shimadzu XRD 6000, Shimadzu corporation, Japan) with Ni-filtered Cu K_α radiation (λ = 0.154060 nm). The experimental procedure were shown in Fig. 1

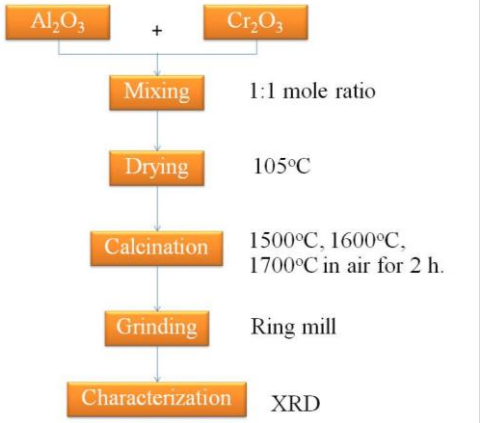

Figure 1 Experimental schematic diagram for preparing Al_2O_3/ Cr_2O_3 solid solution

3. Results and discussion

Phase analysis of calcined powders of Al_2O_3 : Cr_2O_3 at 1:1 mole ratio were characterized by XRD. The XRD patterns of calcined powders at various temperatures from 1,500°C to 1,700°C for 2 h were shown in Fig, 2. The XRD pattern of all calcined temperature illustrated two main phase, Al_2O_3 precursor and $(Cr_{1-x},Al_x)_2O_3$ which $0 \leq x \leq 1$. When calcined temperature was higher, intensity of $(Cr_{1-x}Al_x)_2O_3$ peaks slightly increased and shifted

to Al_2O_3 peaks while intensity of Al_2O_3 peaks gradually decreased. From this result, it can be implied that Cr^{3+} in Cr_2O_3 rhombohedral structure was gradually substituted by Al^{3+} at high temperature. The completed reaction was shown at 1,700°C. In order to confirm shifting of diffraction peak of new phase, the position of the highest peak of $(Cr_{1-x},Al_x)_2O_3$ were shown in Fig. 3. Moreover, new phase formation reaction was predicted as equation (1) [8].

$$Al_2O_3 + Cr_2O_3 \longrightarrow 2(Cr_{1-x}Al_x)_2O_3 \quad ...(1)$$

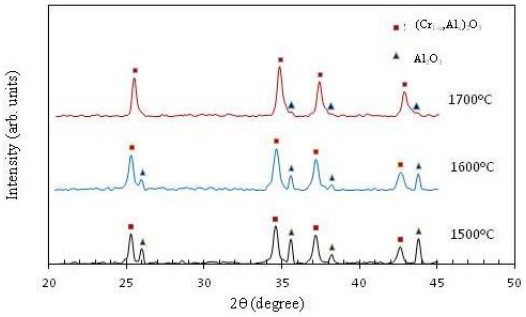

Figure 2 The XRD pattern of calcined samples with different temperatures

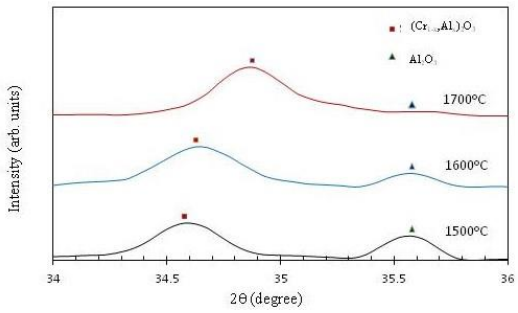

Figure 3 The highest peak position of $(Cr_{1-x},Al_x)_2O_3$ and Al_2O_3

To investigate for each the shifting of peak that depending on temperature, *d*-spacing in 311 plane, lattice parameters (*a* and *c*) were calculated and plotted to observed the changing of lattice parameters depending on temperature.

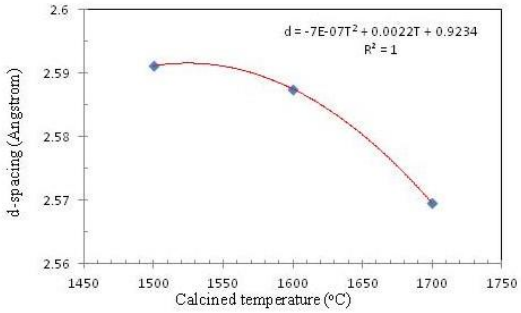

Figure 4 The relationship of calcined temperatures and *d*-spacing in 311 plane

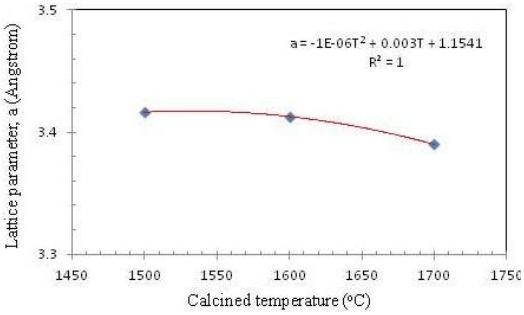

Figure 5 The relationship of calcined temperatures and lattice parameter, *a*

The relationships of calcined temperatures and lattice parameters (*d*-spacing in 311 plane, *a* and *c*) were shown in Fig. 4, Fig. 5 and Fig. 6 respectively. The relationships were predicted by equation (2), (3) and (4) respectively.

$d = -7.0\times10^{-7}T^2 + 2.2\times10^{-3}T + 0.92$ ……. (2)

$a = -1.0\times10^{-6}T^2 + 3.0\times10^{-3}T + 1.15$ ……. (3)

$c = -4.0\times10^{-6}T^2 + 1.3\times10^{-2}T + 6.07$ ……. (4)

Where d = d-spacing in 311 plane (Angstrom)

a = lattice parameter, a (Angstrom)

c = lattice parameter, c (Angstrom)

The lattice parameters are depending on calcined temperature as polynomial squared.

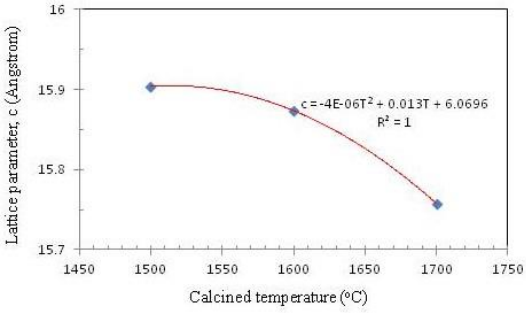

Figure 6 The relationship of calcined temperatures and lattice parameter, c

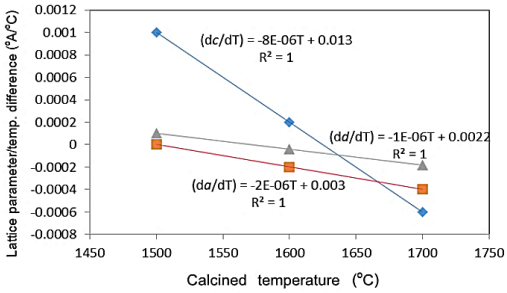

Figure 7 The relationship of calcined temperature and lattice parameter/temperature difference

Lattice parameters/temperature differences are depending on calcined temperature as linear. The relationships were shown by equation (5), (6) and (7) respectively.

$dd/dT = -1.4\times10^{-6}T + 2.2\times10^{-2}$ …… (5)

$da/dT = -2.0\times10^{-6}T + 3.0\times10^{-3}$ …… (6)

$dc/dT = -8.0\times10^{-6}T + 1.3\times10^{-2}$ …… (7)

c/a ratio were plotted in Fig. 8 which depended on the calcined temperatures as linear. The relationships were shown by equation (8).

$c/a = -5\times10^{-5}T + 4.73$ ……..…….. (8)

Lattice parameters inverse with the calcined temperatures, when the calcined temperatures were increasing, lattice parameters were decreasing. From these results showed that the reaction rate of this solid solution was depending on the calcined temperature.

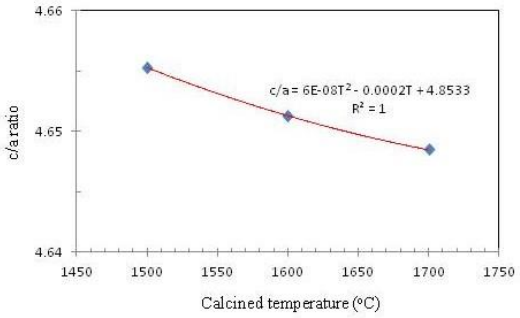

Figure 8 The relationship of calcined temperatures and c/a ratio

4. Conclusions

In this experiment, $(Cr_{1-x},Al_x)_2O_3$ was prepared by solid state reaction which x was depending on the calcined temperature and lattice parameters were inversing with the calcined temperature. In addition, Cr^{3+} in Cr_2O_3 rhombohedral structure was gradually substituted by Al^{3+} at high temperature.

5. References

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